Boletín de la Sociedad Española de Cerámica y Vidrio
Vol 52, 1, 42-47, Enero-Febrero 2013 ISSN 0366-3175. eISSN 2173-0431.
doi: 10.3989/cyv.52013

BOLETIN DE LA SOCIEDAD ESPAÑOLA DE
Cerámica y Vidrio
A R T I C U L O

An investigation of pottery production technology for the *West Slope* wares from Dorylaion (Eskişehir/Turkey)

A. İSSİ[1], A. KARA[2]

[1]Department of Materials & Ceramic Engineering, Dumlupınar University, Kütahya, Turkey
[2]Department of Materials Science and Engineering, Anadolu University, Eskişehir, Turkey

Researches in the field of pottery production technology in ancient times, done in different settlements during the same period, may contribute to know relationships established within the different cultural communities. In ancient times, Anatolia (Asia Minor) was the crossroads of ancient civilizations. There are several ancient settlements and artifacts belonging to Hellenistic culture (330-30 BC). West Slope wares from Dorylaion (Eskişehir/Turkey) excavations are the main Hellenistic culture findings. In this study, different analytical techniques were employed for the characterization of these findings in order to enlighten the pottery production technology. Wavelength dispersive X-ray fluorescence (WDXRF) and X-ray diffraction (XRD) were employed to study the chemical and mineralogical composition of the bodies, respectively. Scanning electron microscopy (SEM) and energy dispersive X-ray spectrometry (EDX) were also performed for the microstructural and microchemical characterization of body and slip layers of the selected potsherds. The raw materials used, firing temperatures and atmosphere and related microstructural characteristics were discussed.

Key words: West Slope Ware, Characterization, Microstructure, Archaeometry, Ceramic.

Investigacion sobre la tecnologia de producción de utensilios tipo "West Slope" procedentes de Dorylaion (Eskişehir/ Turquía)

La investigación de la tecnología utilizada por distintas civilizaciones en la producción de piezas cerámicas encontradas de un mismo asentamiento arqueológico, puede contribuir al conocimiento de relaciones establecidas entre distintas comunidades culturales. Anatolia (Asia Menor) es considerada una importante encrucijada de antiguas civilizaciones que abarcan del Periodo de Bronce al imperio Otomano. Las piezas conocidas como *West Slope* son los principales restos de la cultura helenística (330-30 A.C.) encontrados en las excavaciones de Dorylaion (Eskişehir/ Turquía). En este estudio, diferentes técnicas analíticas han sido empleadas en la caracterización de estas piezas, a fin de dilucidar la tecnología utilizada en su produción.. Fluorescencia de rayos X por onda dispersa (WDXRF) y difracción de rayos X (XRD) fueron empleados para estudiar las composición elemental y mineralógica de las piezas, respectivamente. Microscopia electrónica (SEM) y espectrometría de rayos X de energía dispersiva (EDX) fueron a su vez utilizadas para la caracterización de microquímica y microestructural de los fragmentos seleccionados. Las materias primas utilizadas, temperaturas y atmósfera de cocción, y las características microestructurales son discutidas en este trabajo.

Palabras clave: Artículos occidentales, Caracterización, Microestructura, Arqueometría, Cerámico.

1. INTRODUCTION

Pottery production in ancient times was widespread due to the ease of production than metallic wares and being owned easily by ordinary people. Production of black and glossy West Slope wares may be considered an imitation facility of metallic wares in Hellenistic period (330-30 BC). "West Slope" ware is named from early findings made on the west slope of the Athenian Acropolis. It is a class of Hellenistic pottery that is essentially black glazed with relief and polychrome decoration. Probably, these wares were very fashionable cups within the Mediterranean basins during the Hellenistic period [1-3].

There were important Hellenistic settlements in Anatolia (Asia Minor) during that period. Dorylaion was one of them. Dorylaion is located about 3 km north-east of Eskişehir (Turkey) and it is called Şarhöyük today (Figure 1). Dorylaion was at the junction of some of the most important roads of the central Anatolia leading to Marmara Sea, Aegean coast and the Mediterranean region. Regular excavations have been carried out at Dorylaion since 1989 by a team from Anadolu University in order to unearth the remnants of several civilizations lived there. These excavations have brought to light a wide variety of material revealed from the cultures of Ottoman, Byzantine, Roman, Hellenistic, Classical Phrygian, Hittite and First Bronze Period.

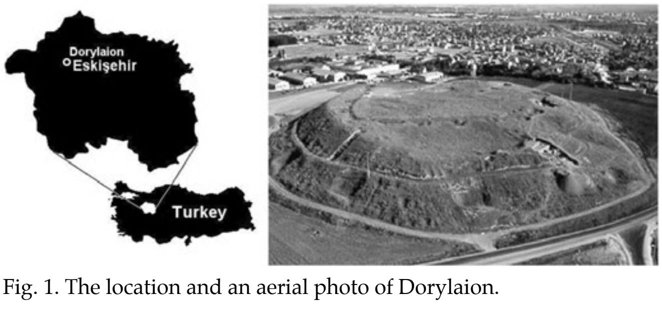


Fig. 1. The location and an aerial photo of Dorylaion.

It is believed that the characterization of West Slope wares found at Dorylaion may contribute to know better pottery production technology and its spreading areas during Hellenistic period. Thus, different analytical techniques were performed for the characterization of West Slope wares. Wavelength dispersive X-ray fluorescence (WDXRF) and X-ray diffraction (XRD) were employed to study the chemical and mineralogical composition of the bodies, respectively. Scanning electron microscopy (SEM) and energy dispersive X-ray spectrometry (EDX) were also performed for the microstructural and microchemical characterization of body and slip layers of the ceramic pieces.

2. MATERIALS AND METHODS

Twenty two West Slope wares were representatively chosen for the study. The images of some selected West Slope wares (designated as B) are shown in Figure 2. Potsherd types are the handles and the rim of the wares. Their colors are orange, red, brown and black. There are also some plants or figurative decorations. The bodies are a buff or light brown color and have a homogeneous fine-grained texture.

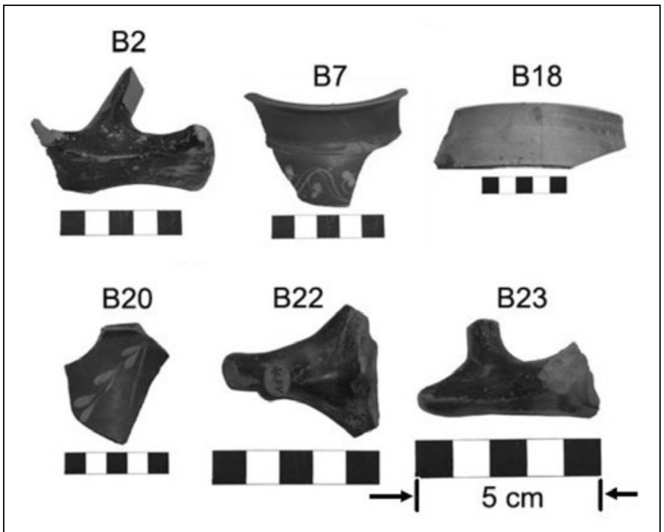


Fig. 2. The images of some selected West Slope wares (designated as B).

Fine powders were prepared in an agate mortar after abrading the slip layers of the potsherds in order to be analyzed by WDXRF and XRD techniques. Rigaku ZSX primus and Rigaku Rint 2200 powder diffractometer with Cu $K\alpha$ radiation was used for WDXRF and XRD analyses, respectively. Glass tablets were prepared by fluxing for the analysis with a ratio of 1/10 powder to $Li_2B_4O_7$ in weight to study chemical analysis of major and minor elements of the potsherds. XRD patterns were obtained by scanning 5^0 to 70^0 2Θ, with a goniometer speed of 2°/min., operating at 40 kV and 30 mA. The bulk samples from the potsherds were cross-sectional impregnated with polymeric resin in vacuum and then polished to obtain flat surfaces to investigate specifically in back-scattered electron (BSE) mode. In order to prevent charging under electron beam, samples were coated with Au/Pd target material. A Zeiss Evo 50EP scanning electron microscopy (SEM) attached with Oxford instruments energy dispersive X-ray spectrometry (EDX) was used for the microstructural and microchemical characterization.

3. RESULTS AND DISCUSSION

3.1. WDXRF Results

There are different analytical techniques for chemical analysis such as optical emission spectrometry (OES), X-ray fluorescence (XRF), neutron activation analysis (NAA), particle induced X-ray emission (PIXE), atomic absorption spectrometry (AAS) and inductively coupled plasma (ICP) [4]. Major, minor and trace elements detected in the samples may be an indicative results for provenance studies. This study focuses on the technological properties of the potsherds. Therefore, WDXRF analyses of the sherds were performed in semi-quantitative scale and trace elements in ppm amount were not considered exactly here. The similarities and differences of the sherds in major and minor scale of elements and relations to mineralogical content were discussed.

Samples have different chemical composition in terms of the raw materials used for their production. The relation to SiO_2 and CaO in the results is important for the indication of siliceous and calcareous mineralogical assemblages used for the bodies.

TABLE I. WDXRF RESULTS OF SOME WEST SLOPE WARES.

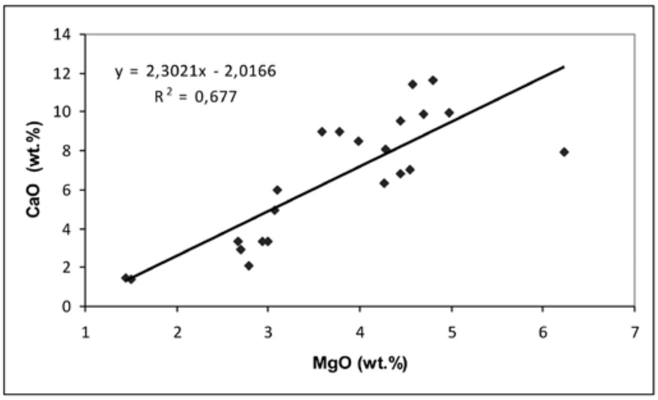
	Experimental code					
	Calcium rich			**Calcium poor**		
Oxide (wt. %)	**B20**	**B7**	**B2**	**B23**	**B22**	**B18**
SiO_2	52.99	52.36	52.14	58.01	58.57	61.85
Al_2O_3	17.38	15.67	16.69	24.84	24.29	19.81
Fe_2O_3	7.96	8.76	7.77	5.79	5.72	6.72
MgO	4.98	4.57	4.80	1.50	1.44	2.79
Na_2O	0.90	0.97	0.94	1.56	1.37	1.53
K_2O	4.41	4.23	4.63	5.49	5.97	3.92
CaO	**9.99**	**11.44**	**11.61**	**1.42**	**1.44**	**2.09**
TiO_2	0.87	1.06	0.83	0.82	0.80	0.90
P_2O_5	0.15	0.15	0.20	0.08	0.09	0.09
SO_3	0.02	0.05	0.04	0.02	0.02	-
Cr_2O_3	0.05	0.08	0.05	-	-	-
MnO	0.13	0.15	0.11	0.10	0.13	0.14
NiO	0.04	0.04	0.04	-	-	0.02
CuO	0.02	0.03	0.02	0.02	0.02	0.02
ZnO	0.01	0.03	0.04	0.03	0.03	0.02
As_2O_3	-	-	-	0.01	-	-
Rb_2O	0.04	-	0.03	0.06	0.05	0.04
SrO	0.03	0.03	0.03	0.02	0.02	0.02
ZrO_2	0.02	0.02	0.02	0.03	0.03	0.03
BaO	-	0.36	-	0.20	-	-

(-) : Not detected or below the detection limit

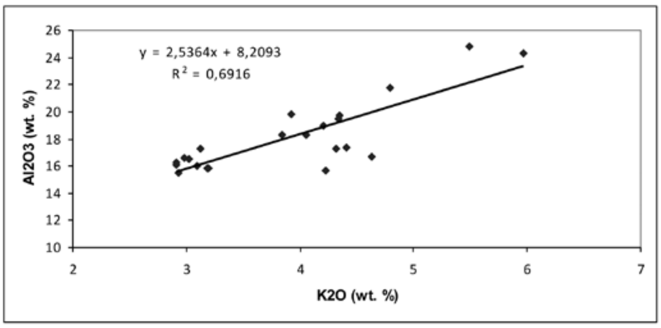
Some of the samples have higher amount of CaO. This indicates that calcareous raw materials were also used for the production besides siliceous raw materials. WDXRF results of selected calcium rich and calcium samples are given in Table I.

CaO changes between 1.42 and 11.61% while MgO changes between 1.44- 6.23%. It may be considered that the source of CaO is not only dolomitic materials but predominantly the calcareous materials. Binary plot (Figure 3a) shows the correlation between CaO and MgO for all the samples. The correlation coefficient (R) is a measure of how much linear relationship exists between the values for the two variables. The coefficient of determination (R^2) is a measure of how well the regression line represents the data. It would be close to 1, if the variance for CaO approached to variance in MgO. The relationship is more evident for Al_2O_3 and K_2O. K_2O usually originates from either feldspar or clay. Both cases require a linear increase or decrease in the amount of Al_2O_3 and K_2O. R^2 for Al_2O_3 and K_2O is stronger than the relationship between CaO and MgO (Figure 3b).

K_2O mass percent varies between 2.91 - 5.97%. It changes in moderate range while Na_2O varies between 0.73 - 2.28%. These alkali contents should be supplied by especially feldspathic contents, principally K-feldspars. As known, alkaline and earth alkaline oxides may act as fluxes and promote neo-mineral formation at lower sintering temperatures, respectively. The highest total alkaline and earth alkaline amounts were found in B2 sample. Iron oxide, expressed as Fe_2O_3, changes from 5.72 to 12.91% and it should be the principal colorant for the bodies.



(a)



(b)

Fig. 3. Binary oxide plots showing the correlation between (a) CaO and MgO and (b) Al_2O_3 and K_2O for all the samples.

3.2. XRD Results

XRD results showed different mineralogical contents. Identified minerals are quartz (SiO_2), plagioclase [(Na,Ca) $AlSi_3O_8$], alkali feldspar ($KAlSi_3O_8$), illite/muscovite [(K,H_3O) $Al_2Si_3AlO_{10}(OH)_2$]/[(K,Na)(Al,Mg,Fe)$_2$($Si_{3.1}Al_{0.9}$)$O_{10}(OH)_2$, potassium mica ($KAl_3Si_3O_{11}$), magnesiohornblende [(Ca,Na)$_{2.26}$(Mg,Fe,Al)$_{5.15}$(Si,Al)$_8O_{22}(OH)_2$], anorthoclase [(Na,K)(Si_3Al) O_8], lepidocrocite [FeO(OH)], hematite (Fe_2O_3), hercynite ($FeAl_2O_4$), calcite ($CaCO_3$), gehlenite ($Ca_2Al_2SiO_7$), diopside/augite [Ca(Mg,Al)(Si,Al)$_2O_6$]/[Ca(Mg,Fe)Si_2O_6]. The absence or presence some minerals/phases in the bodies are related to the temperature exposed. Some relevant studies in the literature may help to estimate the firing temperature of a potsherd. Structure of clay minerals illite/muscovite break down in the range of 900-1000^{0}C [5-8]. If the firing temperature does not exceed this range, these minerals still continue to exist as components of the body. For further stages of firing temperature, from destruction of illite, an intermediate phase between spinel (MgO.Al_2O_3) and hercynite (FeO.Al_2O_3) originates [9]. The formation of spinel phases is affected by the presence or absence of interlayer cations, substitutional impurities and accessory minerals in clays [10]. Calcite decomposition into CaO and CO_2 begins around 750^{0}C and it is completed up to a temperature of 900^{0}C. Although, Trindade et. al. concluded that decarbonation of calcite may extend to 1100^{0}C for calcite rich systems [11]. However, secondary calcite may take place in ceramics as a result of post-burial deposition processes due to recarbonation of lime [5]. Dolomite decomposition begins around 650^{0}C into calcite and MgO. Identification of calcite as a transient phase at this stage is, however, difficult by conventional diffraction methods [12]. Decarbonation of dolomite is completed up to 750-800^{0}C [7]. CaO is a highly reactive oxide and reacts with silica and forms wollastonite around 850^{0}C. Gehlenite may be formed at 850^{0}C with the reaction of CaO and illite. Pyroxenes like diopside and augite may be generated from dolomite and silica reactions at 800-900^{0}C [13]. Moreover, these neo-mineral formation temperatures may be affected by firing type such as pit or kiln firing. For instance; calcite decomposition ends around 825^{0}C in kiln firing but it tends to 875^{0}C in pit firing conditions [14]. Quartz and feldspars are the most abundant residual components of such kind of products which can persist up to 1000^{0}C [15]. These reactions, of course, are related to temperature, soaking time at peak temperature, abundance and the type of minerals/phases present, atmosphere, pressure, specific surface area of components, and, etc. The results of XRD and estimated firing temperatures of the potsherds are given in Table II.

Group A is a cluster of illitic/micaceous and iron rich siliceous clay contents. Group B differs from group A with its calcareous content and group C with its dolomitic content. Group BC is a mixture of these groups. But, group D with its only one sample, B8, has different mineralogical assemblages from the others. The subgroups were generated from the mineralogical assemblage difference in the groups and there is a tendency for higher firing temperature requirement between the subgroups. For example, the difference between BC1 and BC2 originates from the absence of dolomite in the subgroup BC2. Dolomite first decomposes into calcite and MgO, as previously expressed. The other minerals/phases are the same. Calcite in the subgroup BC2 may be

Bol. Soc. Esp. Ceram. Vidr. Vol 52. 1, 42-47, Enero-Febrero 2013. ISSN 0366-3175. eISSN 2173-0431. doi: 10.3989/cyv.52013

an end product of dolomite decomposition. Therefore, the subgroup BC2 requires relatively higher firing temperatures than BC1 for its minerals/phases. It should also be noticed that estimation of firing temperatures were evaluated as the maximum temperatures reached during firing for the potsherds considering the presence of new mineral/phase formations and excluding the presence of some trace amounts of primary minerals such as dolomite in C and BC groupings. This should be the reason of different firing temperatures reached inside the potsherds during firing [16].

Fragments with clay minerals should not have exceeded 900-950^{0}C while calcitic or dolomitic bodies have lower temperatures of 850-900^{0}C for the decarbonation. Pieces without clay minerals and carbonated minerals should have reached the temperatures of 900-1000^{0}C. The bodies with clay minerals and/or carbonated minerals have been inferred to lower temperatures in a range of 600-700^{0}C. The decomposition of carbonated minerals was confirmed with TG-DTA analyses. The results of TG-DTA analyses supplied some information to firing temperatures but these techniques always need other comparative results.

Lepidocrocite readily transforms into hematite around the temperature of 500^{0}C [17]. For this reason, sample coded as D1 may be concluded as it was fired in the lowest firing temperature. Hematite, which was observed in almost all the body samples, may be considered as an indication that firing atmosphere was finalized in oxidative condition.

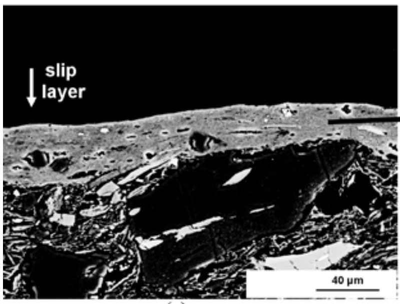
Clay deposits may include some non-plastic materials like quartz, feldspars and iron compounds. Non-plastic materials may be natural compounds of the clays or they may be used as additional materials. The addition of temper materials such as siliceous raw materials, shells or crushed potsherds may be found in several artifacts [15]. Temper materials may improve the mechanical properties of the pots during both shaping and firing processes. Especially, calcareous materials may contribute to preserve the desired shape after forming [18]. All the potsherds do not have any calcareous materials. Probably, the change inside or between the clay deposits should have determined the type of raw materials used for production.

3.3. SEM-EDX Results

Microstructural characteristics were examined with SEM in order to obtain detailed information. Chemical

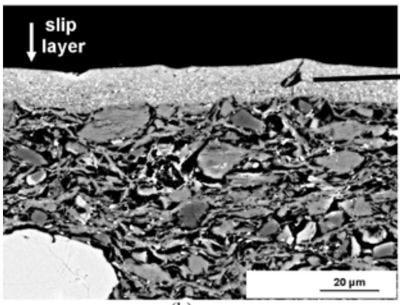
TABLE II. XRD RESULTS OF THE POTSHERDS AND ESTIMATED FIRING TEMPERATURES.

Group	Code	Minerals/Phases	Experimental code	Estimated firing temperature (^{0}C)
A	A1	quartz, alkali feldspar, plagioclase, illite/muscovite, hematite	B1	600-700
	A2	quartz, alkali feldspar, plagioclase, potassium mica, illite/muscovite, hematite	B23	600-700
	A3	quartz, alkali feldspar, plagioclase, hematite	B3, B5, B6, B11, B12, B18	900-1000
	A4	quartz, alkali feldspar, plagioclase	B9	900-1000
	A5	quartz, alkali feldspar, plagioclase, hercynite, hematite	B22	1000-1050
B	B1	quartz, alkali feldspar, plagioclase, calcite, illite/muscovite, hematite	B10	600-700
C	C1	quartz, alkali feldspar, plagioclase, dolomite, diopside/augite, illite/muscovite, hematite	B16	850-900
	C2	quartz, alkali feldspar, anorthoclase, plagioclase, diopside/augite, hematite	B4, B7	900-1000
BC	BC1	quartz, alkali feldspar, plagioclase, dolomite, diopside/augite, gehlenite, illite/muscovite, hematite	B2	800-850
	BC2	quartz, alkali feldspar, plagioclase, calcite, diopside/augite, gehlenite, illite/muscovite, hematite	B13, B15	800-850
	BC3	quartz, alkali feldspar, plagioclase, dolomite, diopside/augite, gehlenite, illite/muscovite, hematite	B17	800-850
	BC4	quartz, alkali feldspar, plagioclase, diopside/augite, gehlenite, hematite	B14	900-1000
	BC5	quartz, alkali feldspar, anorthoclase, plagioclase, diopside/augite, gehlenite, hematite	B21	900-1000
	BC6	quartz, alkali feldspar, anorthoclase, plagioclase, diopside/augite	B20	900-1000
D	D1	quartz, alkali feldspar, plagioclase, magnesiohornblende, illite/muscovite, lepidocrocite, hematite	B8	<500



(a)

B8	
Oxide	wt. %
SiO_2	49.62
Al_2O_3	31.29
Fe_2O_3	7.48
MgO	1.69
Na_2O	3.34
K_2O	5.28
CaO	0.98
TiO_2	0.32



(b)

B23	
Oxide	wt. %
SiO_2	43.28
Al_2O_3	30.84
Fe_2O_3	20.92
MgO	1.42
Na_2O	0.42
K_2O	2.37
CaO	0.16
TiO_2	0.59

Fig. 4. Back-scattered electron (BSE) images of (a) B8, (b) B23 and semi-quantitative EDX results of the slip layers.

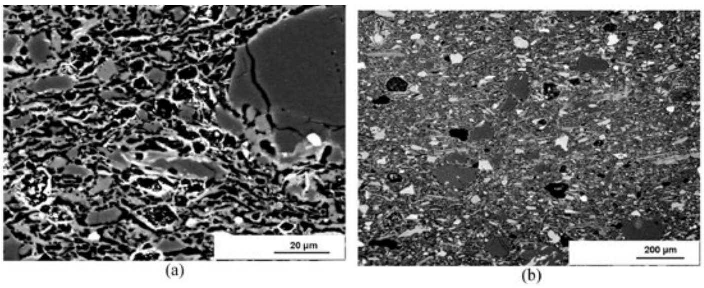


Fig. 5. (a) BSE image showing progressive welding behavior between clay matrix and grains related to temperature of firing of B4 sample and (b) BSE image of B8 sample deduced with a low firing temperature.

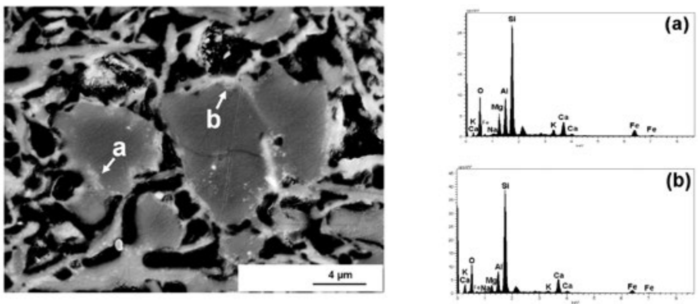


Fig. 6. BSE image of neo-mineral formation of pyroxenes for B20 sample.

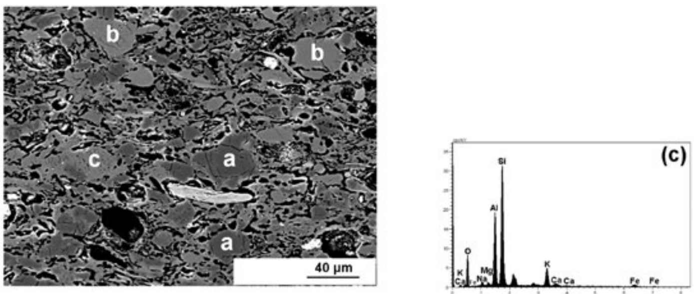


Fig. 7. Some of the firing temperature effects on the microstructure of B11 sample.

compositions of the body and slip layers were also analyzed by EDX. In general, EDX results of the bodies were similar to WDXRF results in the composition of major and minor elements with a predictable deviation caused by inhomogeneous grain distribution on the cross sectional area of the potsherds investigated. Slip layers principally have an iron rich composition with different elements. The minimum and maximum iron oxide amounts in weight (%) are in the samples designated B8 and B23, respectively. Back-scattered electron (BSE) images of B8 and B23 and semi-quantitative EDX results of their slip layers are given in Figure 4 (a) and (b).

The slip layers have a dense microstructure and are easily distinguished from the body layer of the potsherds. Some slip layers include pore structures inside. These pores should have been originated from the use of organic materials in slip batches. There are also some residual minerals inside the slip layers. Iron oxide was expressed as Fe_2O_3 for the EDX results. Its amount changes between 7.48-20.92% for the slip layers. The type of iron oxide determines the color of the slip layer. Hematite, maghemite and magnetite minerals in the slip layer and their characteristics were discussed in a previous study [19]. K_2O is higher than the other alkali and earth alkali elements. It indicates a typical iron rich clayey batch. Pottery fragments should have been buried more than 2000 years, but they are still durable. Slip layers should have principally similar thermal expansion coefficients to the body layers since almost all the slip layers have no cracks or spalling. The thickness of slip layers change between from 5 to 40 μm. They might have been applied to body surfaces in the form of suspension. They have nearly the same elemental composition but different in colors. It shows that the redox conditions of the kilns in which they were fired could be adjusted [20, 21].

Some interactions occur in the structure of clay based systems with the increasing temperature. Some of them are welding between clay matrix and mineral grains, shape changes of mineral phases, an increase of the aggregation rate within the clay matrix with the formation of secondary porosity and intergranular bridges [22]. BSE images of the potsherds at different magnifications concluded the effects of firing on the microstructures. Atomic contrast both between the matrix components and at the rims of the grains can be noticed easily in this mode. It is evident that there is a progressive welding behavior between clay matrix and grains related to temperature of firing of B4 (Figure 5 (a)). Rounded pores may also be assigned as the indication of the vitrification degree in the microstructure. Lower sintering temperature should have not made an impressive change such as the microstructure of B8 sample (Figure 5 (b)).

Small crystallites of Ca, Mg and Fe rich alumina silicates observed at higher magnifications in a siliceous grain in Figure 6 and at the rim of another siliceous grain may be inferred as the neo-mineral formation of pyroxenes related with firing temperature reached for B20 sample.

B11 sample was believed to be made of siliceous clay. Some of the effects of firing temperature observed in Figure 7 are the intergranular bridges between both quartz (a) and feldspar (b) grains. EDX spectrum from the grain indicates a multi elemental composition (Figure 7 (c)). This constitution should be a new grain formation from partially vitrified matrix components.

Bol. Soc. Esp. Ceram. Vidr. Vol 52. 1, 42-47, Enero-Febrero 2013. ISSN 0366-3175. eISSN 2173-0431. doi: 10.3989/cyv.52013

4. CONCLUSIONS

The West Slope wares from Dorylaion were produced with different clays including siliceous, calcareous, dolomitic and also a mixture of these raw materials. They probably were applied with an iron rich clayey suspension. These ceramic wares were exposed to different firing temperatures. Different colors among slip layers concluded that the firing atmosphere could be adjusted. Black and glossy wares without cracks or spalling showed that Hellenic potters were experienced in production. These results and potsherd characteristics need to be compared with other Hellenistic settlements with a view to understand the scale of pottery production technology during the Hellenistic period.

ACKNOWLEDGEMENTS

This study was financially supported by the Scientific and Technological Research Council of Turkey (TÜBİTAK) with the project number 106M463. We would like to thank also Prof. Dr. Taciser Sivas and Assoc. Prof. Dr. Hakan Sivas from Anadolu University/Turkey for the archaeological samples and their contributions to the introduction of Hellenistic wares.

REFERENCES

1. C. Watzinger, Vasenfunde aus Athen, p. 70-84, Mitteilungen des Deutschen Archaologischen Instituts, Berlin, 1901 (in German).
2. F.F. Jones, Excavations at Gozlu Kule, Tarsus, The Hellenistic and Roman period, p. 149-296, Vol. 1, In: H. Goldman (Ed), Princeton University Press, Princeton, 1950.
3. A. Conze, Altertümer von Pergamon, Deutsches Archäologisches Institut, Vol.1-2, Berlin, 1913 (in German).
4. A.E. Pillay, C. Punyadeera, L. Jacobson, J. Eriksen, Analysis of ancient pottery and ceramic objects using X-ray fluorescence spectrometry, X-Ray Spectrometry 29 53-62 (2000).
5. C. Papachristodoulou, A. Oikonomou, K. Ioannides, K. Gravani, A study of ancient pottery by means of X-ray fluorescence spectroscopy, multivariate statistics and mineralogical analysis, Analytica Chimica Acta 573 347-353 (2006).
6. R.W. Grimshaw, The Chemistry and Physics of Clays and Allied Ceramic Materials, p. 727, Techbooks, India, 1971.
7. L. Maritan, C. Mazzoli, L. Nodari, U. Russo, Second Iron Age grey pottery from Este (northeastern Italy): study of provenance and technology, Applied Clay Science 29 31-44 (2005).
8. J.M. Bhatnagar, R.K. Goel, Thermal changes in clay products from alluvial deposits of the Indo-Gangetic plains, Construction and Building Materials 16 113-122 (2002).
9. M.M. Jordan, A. Boix, T. Sanfeliu, C. de la Fuente, Firing transformations of Cretaceous clays used in the manufacturing of ceramic tiles, Applied Clay Science 14 225-234 (1999).
10. C.J. McConville, W.E. Lee, Microstructural development on firing illite and smectite clays compared with that in kaolinite, Journal of the American Ceramic Society, 88 2267–2276 (2005)
11. M.J. Trindade, M.I. Dias, J. Coroado, F. Rocha, Mineralogical transformations of calcareous rich clays with firing: A comparative study between calcite and dolomite rich clays from Algarve, Portugal, Applied Clay Science 42 345-355 (2009).
12. A.H. De Aza, X. Turrillas, J.L. Rodríguez, P. Pena, Estudio del proceso de sinterización reactiva en sistemas con dolomita mediante termodifractometría de neutrones, Boletin de la Sociedad Espanola de Ceramica y Vidrio, 43 [1] 12-15 (2004).
13. G. Cultrone, C. Rodriguez-Navarro, E. Sebastian, O. Cazalla, M.J. De La Torre, Carbonate and silicate phase reactions during ceramic firing, European Journal of Mineralogy 13 621-634 (2001).
14. L. Maritan, L. Nodari, C. Mazzoli, A. Milano, U. Russo, Influence of firing conditions on ceramic products: Experimental study on clay rich in organic matter, Applied Clay Science 31 1-15 (2006).
15. A. Iordanidis, J. Garcia-Guinea, G. Karamitrou-Mentessidi, Analytical study of ancient pottery from the archaeological site of Aiani, northern Greece, Materials Characterization 60 292-302 (2009).
16. M. Maggetti, Ch. Neururer, D. Ramseyer, Temperature evolution inside a pot during experimental surface (bonfire) firing, Applied Clay Science, 53 500-508 (2011)
17. T. Glotch, M. Kraft, Thermal transformations of akaganeite and lepidocrocite to hematite: assessment of possible precursors to Martian crystalline hematite, Physics and Chemistry of Minerals 35 569-581 (2008).
18. M.I. Carretero, M. Dondi, B. Fabbri, M. Raimondo, The influence of shaping and firing technology on ceramic properties of calcareous and non-calcareous illitic-chloritic clays, Applied Clay Science 20 301-306 (2002).
19. A. İssi, A. Raškovska, A. Kara, O. Grupce. B. Minčeva-Šukarova, F. Okyar, Scanning electron microscopy and micro-Raman spectroscopy of slip layers of Hellenistic ceramic wares from Dorylaion/Turkey, Ceramics International, 37 1879-1887 (2011)
20. J. van der Weerd, G.D. Smith, S. Firth, R.J.H. Clark, Identification of black pigments on prehistoric Southwest American potsherds by infrared and Raman microscopy, Journal of Archaeological Science 31 1429-1437 (2004).
21. C.C. Tang, E.J. MacLean, M.A. Roberts, D.T. Clarke, E. Pantos, A.J.N. Prag, The study of Attic black gloss sherds using synchrotron X-ray diffraction, Journal of Archaeological Science 28 1015-1024 (2001).
22. M.P. Riccardi, B. Messiga, P. Duminuco, An approach to the dynamics of clay firing, Applied Clay Science 15 393-409 (1999).

Recibido: 22/10/2010
Aceptado: 21/02/2012